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Production of Potassium Hydroxide by Conversion – Extraction Method Based on Local Materials

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Abstract. The aim of the work was to study and develop a technological process for obtaining potassium hydroxide by conversion – extraction method based on local materials. Optimal technological parameters of the process have been established. A number of physico-chemical properties of the resulting product have been investigated depending on the technological parameters. The stages of the basic technological scheme for the production of potassium hydroxide based on lime milk and potash are recommended.

Keywords: lime milk, potassium carbonate, calcium carbonate, potassium hydroxide, caustification, temperature.

Among the identified known [1-6] methods for obtaining and purifying potassium hydroxide, the methods of its purification by co-precipitation are of the greatest interest. Various chemical compounds are used as reagents used to precipitate impurities, so-called collectors, in known methods. As such, for example, calcium carbonate is used in the form of lime, chalk or marble with a particle size of 0.1-5 mm [7], magnesium oxide [8], basic magnesium carbonate. The disadvantages of these purification methods, first of all, are an insufficiently high degree of purification, as well as the inability to purify potassium hydroxide from a significant group of impurities in one process, and not only from iron, calcium and magnesium [9].

A method is known for cleaning an alkali solution from impurities of iron, calcium and magnesium [10]. Purification by this known method is carried out by filtering an alkali solution at room temperature through a layer of basic magnesium carbonate with a height of 3-100 mm. The main disadvantage of the method is the inability to purify potassium hydroxide from several impurities at once in one process. In addition, the known method is not technological and not economical, since the use of columns with a processing agent leads to their rapid contamination and failure [10].

To improve the quality of potassium hydroxide, a method for cleaning its solution is proposed, which includes processing the initial product with basic magnesium carbonate. To purify a solution of potassium hydroxide, basic magnesium carbonate containing 0.1 – 0.5 wt is used.% of lanthanide salts, which are added by stirring to the solution to be cleaned. After that, the reaction mass is subjected to microfiltration. The invention makes it possible to increase the purity of the resulting product. The main difference of this method is the use as a processing agent (collector) of basic magnesium carbonate containing certain amounts of lanthanide salts. The introduction of this additive into the basic magnesium carbonate in combination with other components provides an increase in the purity of the final product for a number of impurities and allows you to expand the number of metal cation impurities removed.

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Experimental data showed the formation of two layers in the products (lower and upper) with an increase in water content.

Table

Solubility isotherms of the system C_2H_5OH-KOH-H_2O at temperatures

№	Ratios $H_2O : C_2H_5OH$	Temperature, °C	K_2O content in the liquid phase, %	η, °C	ρ, г/sm³
1.	100 : 0	- 3	39,55	1,4607	1,504
2.		0	41,11	1,4619	1,510
3.		20	44,35	1,4634	1,518
4.		40	47,94	1,4642	1,522
5.		60	50,10	1,4718	1,560
6		80	52,14	1,4747	1,575
7.	80 : 20	- 3	32,36	1,4427	1,430
8.		0	34,69	1,4391	1,410
9.		20	35,27	1,4446	1,455
10.		40	36,19	1,4449	1,470
11.		60	37,08	1,4570	1,480
12.		80	38,77	1,4668	1,535
13.	50 : 50	- 3	23,50	1,4231	1,350
14.		0	28,78	1,4087	1,240
15.		20	32,07	1,4220	1,340
16.		40	33,09	1,4391	1,410
17.		60	34,23	1,4430	1,440
18.		80	38,10	1,4510	1,475
19.	20 : 80	- 3	23,44	1,3840	1,100
20.		0	23,93	1,3890	1,105
21.		20	25,33	1,3970	1,070
22.		40	26,47	1,4030	1,074
23.		60	28,85	1,4070	1,077

24.		80	30,96	1,4180	1,153
25.	0 : 100	- 3	30,25	1,3950	1,068
26.		0	32,05	1,403	1,092
27.		20	34,36	1,4007	1,097
28.		40	35,97	1,4002	1,073
29.		60	37,11	1,3930	1,064
30.		80	38,28	1,3810	1,055

In our studies, potash (K_2CO_3) synthesized from potassium chloride of JSC "Dehkanabad Potash Plant" and lime obtained by firing limestone of the Jamansai deposit of the Republic of Karakalpakstan in LLC "Kungrad Soda Plant", as well as ethyl alcohol of the qualification "HC" served as the starting materials for obtaining potassium hydroxide.

After the conversion process was completed, alcohol was added to the pulp and the resulting suspension was divided into liquid (KOH alcohol solution) and solid ($CaCO_3$ precipitate) phases on a laboratory filtration unit. The pulp: alcohol ratio was chosen so that the H_2O: alcohol ratio was within 0.25.

According to the table, the refraction ranges from 1.3810-1.4747. With an increase in temperature, it increases by 0.0010-0.0020, and with a decrease in the ratio of H_2O: alcohol, it decreases. For example, at the ratios H_2O: alcohol 50:50 and 20:80 and a temperature of 40 ^oC, the refraction is 1.4030 and 1.4070, respectively (Table).

The analysis of the chemical composition of the upper (B.c) and lower (N.c) layers shows that the content of CaO and CO_2 in the lower layer is greater than in the upper one.

Therefore, it is desirable to increase the ratio of B.c : N.C., and this is achieved by maintaining the ratio of H_2O : alcohol less than 0.25.

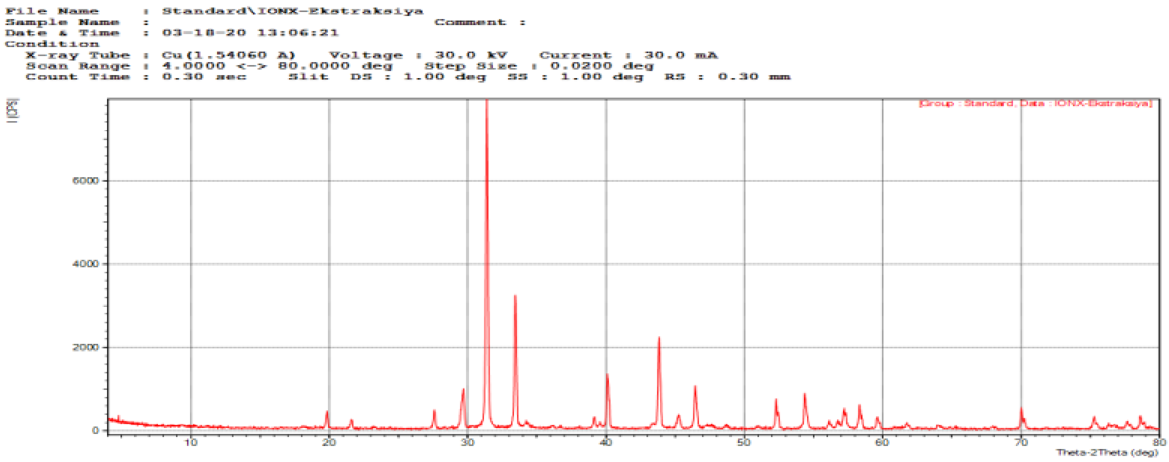
Thus, the optimal composition of the upper layer is, wt%: C_2H_5OH-59.5-61.0; KOH-27.5-33.0; H_2O – 6.0-13%.

It was also found that the resulting calcium carbonate precipitates quickly from the suspension and is easily filtered, which makes it possible to recommend the use of existing standard thickeners and filtration units with a minimum working surface, after which the filtrate is easily evaporated under dilution to 50-80% with further extraction of potassium hydroxide with ethyl alcohol. After distillation, potassium hydroxide was obtained that meets the requirements for products of reactive qualification and special purpose.

A technological scheme is proposed consisting of the stages: conversion, settling, filtration of the condensed part with three-fold washing and return of the washing water to the caustification stage and evaporation of the filtrate to obtain a high concentration KOH solution (40-50%) with its further processing into scaly or tableted potassium hydroxide.

Based on the above, it can be concluded that the optimal parameters of the caustification process are: temperature not less than 95 °C, H_2O ratio:K_2CO_3:$Ca(OH)_2$ - (1.8–3):2:1; the duration of the conversion is at least 120 minutes.

Extraction must be carried out at a ratio of pulp: alcohol 1.0 – 2.0. At the same time, the yield of K_2O in the product is at least 60%, and its concentration reaches at least 45%, resulting in a purified solution of potassium hydroxide.



Pict. Radiograph of precipitation after extraction.

X-ray phase analysis (fig.) showed that the precipitate after extraction contains mainly the following compounds: $CaCO_3$ – 3.012, 2.28, 1.609A°, potassium hydroxide monohydrate ($KOH*H_2O$)-2.85; 1.96; 1.748; 1.04 A°, as well as residues of the initial components $Ca(OH)_2$ – 2.67; 1.95; 1.68 A° and K_2CO_3–2.85; 2.67; 2.06; 1.95; 1.68 A°. Lower filtration rate and reduced K_2O yield in the product are associated with low values of T:W and degree of caustification, respectively.

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